

BSc (Hons) Chemistry

Practical

5th Sem. Paper DSE 2 P

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3. SOIL ANALYSIS

Chemical analysis is generally done under several subject headings :

A. pH of soil (as suspension in water) :

- (a) By matching colour of a piece of pH paper dipped in a water suspension of a soil sample with standard colours of a chart of pH paper.
- (b) Using a pH-meter.

pH of a sample of soil (as filtered water suspension) sample is generally used by using a pH-meter filled with glass electrode.

Required apparatus and chemicals :

- (i) pH-meter & salt -bridge
- (ii) Buffer tablets of pH 4 and 7
- (iii) 40% KCl solution.
- (iv) Finely powdered sample of the soil to tested kept tightly in a small glass bottle, so that it does not above gases like ammonia, HCl, H₂S etc.

Method :

1. The glass electrode and the standard calomel electrode attached property to the pH-meter is connected to each other through a KCl salt bridged and the glass electrode is dipped in distilled water taken in a 100 mL beaker for a few minutes.
2. The two buffer tablets are dissolved in separate volumetric flasks in appropriate volumes of distilled water and buffer solution of pH 4 and pH 7 are prepared.
3. The glass electrode of the pH-meter is then dipped in the buffer first in the solution of pH 4 and next in the solution of pH 7 and the pH-meter is calibrated by adjusting the meter to pH 4 and 7 respectively.
4. The buffer solutions are poured out in respective flasks and the glass electrode is washed with distilled water.
5. 20 g of the powdered soil sample is poured in a conical flask containing 100 mL distilled water and frequent shaken for 1 hr to prepare a 1 : 5 suspension of soil in water. It is filtered.
6. pH of this filtrate is now measured by dipping the glass electrode in it. The electrode is shaken and the reading of the pH-meter is noted when the reading becomes steady. That reading is the pH of the soil.

\therefore % of active carbon in the given soil $\equiv \frac{V \times 1}{0.5} \times 100 \equiv 200 V$ mL 1(N) $K_2Cr_2O_7$ soln.

5. DETERMINATION OF DISSOLVED OXYGEN IN WATER :

Dissolved Oxygen level (D.O.) in natural water and waste water is a very important parameter. D.O. level data of a water body, effluents from a factory and domestic waste water

is extremely important. These are absolute prerequisites of water pollution control and planning of waste treatment processes. Of the different methods available for determination of D.O. in water. **Winkler's method** or iodometric method is most widely used and will be described here.

Chemical principle : Dissolved oxygen in a sample of water is allowed to react with a Mn (II) salt like MnSO_4 in a strongly alkaline medium, when Mn^{+2} reacts with dissolved oxygen and is oxidised to MnO_2 :



This MnO_2 is allowed to react with KI in acid medium, leading to the liberation of iodine which is then titrated with a standard sodium thiosulphate soln.



Calculation :

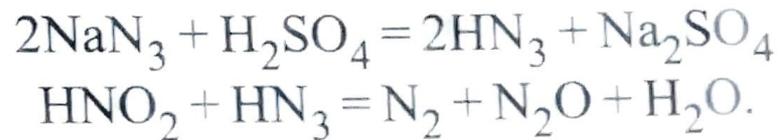


or, $2 \times 1000 \text{ mL } 1(\text{N}) \text{ Na}_2\text{S}_2\text{O}_3 \equiv 16 \text{ g D.O.}$

$\therefore 1 \text{ mL } 1(\text{N}) \text{ Na}_2\text{S}_2\text{O}_3 \text{ soln.} \equiv 0.008 \text{ g D.O.}$

Removal of interference due to the presence of other oxidising agents like NO_2^- in water.

Addition of sodium azide (NaN_3) can remove interference due to NO_2^- . Sodium azide (NaN_3) reacts with NO_2^- in acid medium and decomposes it.



Reagents :

1. 40% NaF or KF solution (to mask Fe^{+3})
2. 36% MnSO_4 solution.
3. Alkaline azide solution prepared by dissolving 50 g NaOH, 13.5 g NaI and 1.0 g NaN_3 in water and diluting to 1 litre.
4. 12(N) H_2SO_4 (1 : 2 conc. H_2SO_4 : water).
5. Solid KI.
6. 0.025 (N) $\text{Na}_2\text{S}_2\text{O}_3$ soln.

Procedure :

Take 100 mL sample water in a 250 mL conical flask and add (i) 2 mL 36% MnSO_4 soln, (ii) 2 mL alkaline azide solution and shake well. Allow the precipitate to settle. Add 6 mL of 12(N) H_2SO_4 and shake the mixture till the precipitate dissolves completely. (iii) Add 0.5-1 g solid KI and dissolve it by shaking. Add 1 mL of starch indicator and titrate with a standard 0.025 (N) $\text{Na}_2\text{S}_2\text{O}_3$ soln. till the pale blue colour becomes colourless by the addition of 1 drop $\text{Na}_2\text{S}_2\text{O}_3$ solution.

Calculation :

We have seen previously that 1(N) $\text{Na}_2\text{S}_2\text{O}_3$ soln. is $\equiv 0.008$ g D.O. Let the volume of $x(\text{N})$ $\text{Na}_2\text{S}_2\text{O}_3$ solution reqd. for titration = V mL.

$\therefore V$ mL $x(\text{N})$ $\text{Na}_2\text{S}_2\text{O}_3$ soln. = $(V \times x)$ mL 1(N) $\text{Na}_2\text{S}_2\text{O}_3$ soln.

\therefore D.O. in 100 mL of water sample = $(V \times x \times 0.008)$ g.

\therefore D.O. in 1 litre of sample water = $(V \times x \times 0.008 \times 10)$ g.

Determination of Biochemical Oxygen Demand (B.O.D.) of a water sample :

The Biochemical Oxygen Demand (B.O.D.) of a sample of water (normal or polluted) is the quantity of oxygen required for the biological decomposition of dissolved organic matter to take place under standard condition in a standard time and temperature. Normally, the time is taken as 5 days and the temperature is 20°C .

Principle : The water sample is taken in an airtight bottle and incubated at 20°C for 5 days. The dissolved oxygen (DO) content of the sample is determined before incubation and after incubation for 5 days at 20°C and B.O.D. is calculated from the difference between initial and final values of DO.

Procedure : DO evaluation is done in both cases (before and after incubation of 5 days at 20°C) by Winklers method described before.

Calculation.

Temperature of the experiment = 20°C

Time of incubation = 5 days

Experimentally found D.O. value by Winkler method.

Before incubation = $(D.O.)_{\text{initial}}$ mg/L

After 5 days incubation = $(D.O.)_{\text{final}}$

Value of B.O.D. = $(D.O.)_{\text{initial}} - (D.O.)_{\text{final}} = \text{B.O.D. m/g}$.

N.B. 1. Drinking water normally has a B.O.D. of less than 1 mg/L

2. Ordinary domestic sewage may have a B.O.D. of 200 mg/L. Any effluent to be discharged into natural bodies (rivers, lakes etc.), of water should have B.O.D. of 200 mg/L or less.

3. It is the only parameter which gives an idea of the biodegradability of any sample of water and self-purification capacity of rivers and streams.

6. DETERMINATION OF CHEMICAL OXYGEN DEMAND (C.O.D.) OF A SAMPLE OF WATER :

Chemical oxygen demand (C.O.D.) of a water sample is a measure of its organic matter-content, and it supplies a very useful information about different water-bodies like lakes, industrial effluents and domestic wastes. Knowledge of C.O.D. is a very important prerequisite for choosing the method of treatment of waste water plants.

Chemical principle :

The principle underlying this method is based on the oxidation of materials present in water by the oxidant potassium dichromate in 50% sulphuric acid, in presence of a catalyst like Ag_2SO_4 and HgSO_4 . Organic matter present in water is oxidised by dichromate and the excess dichromate left is titrated with a standard Mohr salt solution.

Reagents :

1. Standard solution [0.25 (N)] of $\text{K}_2\text{Cr}_2\text{O}_7$ prepared in 18(N) H_2SO_4 .
2. 18(N) sulphuric acid (1 : 1 conc. H_2SO_4 : water)
3. Ag_2SO_4 (solid)

4. HgSO_4 (solid)
5. 0.1 (N) Mohr salt solution prepared in 8(N) H_2SO_4 .
6. Ferroin indicator (1.485 g orthophenanthroline + 0.695 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, dissolved in 100 mL water).

Procedure :

Take 50 mL water sample in a 250 mL conical flask with a standard ground glass joint, add 25 mL 0.25 (N) $\text{K}_2\text{Cr}_2\text{O}_7$ soln. in 18(N) H_2SO_4 , 1 g Ag_2SO_4 and 1 g HgSO_4 .

Reflux the mixture for six hours after fitting a reflux condenser to the conical flask, cool the mixture to room temperature, add 8-10 drops of ferroin indicator and titrate with 0.1 (N) Mohr salt solution in 8(N) H_2SO_4 . At the end-point the colour of the solution changes from light blue to red by the addition of one drop of Mohr salt soln.

Calculation :

Let the volume of the standard $\text{K}_2\text{Cr}_2\text{O}_7$ soln. taken for the experiment is V mL.

Strength of the $\text{K}_2\text{Cr}_2\text{O}_7$ soln. = x (N).

Vol. of Mohr salt soln. reqd. to titrate the excess of $\text{K}_2\text{Cr}_2\text{O}_7$ soln. left after oxidation = V_2 mL.

Strength of Mohr salt soln. = y (N).

$\therefore V_2$ mL of y (N) Mohr salt soln. = $(V_2 \times y)$ mL 1 (N) Mohr salt soln.

$\equiv (V_2 \times y)$ mL 1(N) $\text{K}_2\text{Cr}_2\text{O}_7$ soln.

So, volume of $\text{K}_2\text{Cr}_2\text{O}_7$ soln. left after chemical oxidation of organic material in water = $(V_2 \times y)$ mL of 1(N) $\text{K}_2\text{Cr}_2\text{O}_7$ soln.

\therefore Volume of 1(N) $\text{K}_2\text{Cr}_2\text{O}_7$ solution consumed for oxidation = $[(V \times x) - (V_2 \times y)] = V_1$ mL (say).

Now, 1000 mL of 1(N) $\text{K}_2\text{Cr}_2\text{O}_7$ soln. \equiv 1 eq. wt. of oxygen = 8 g of oxygen.

\therefore 1 mL of 1(N) $\text{K}_2\text{Cr}_2\text{O}_7$ soln. \equiv 0.008 g of oxygen.

$\therefore V_1$ mL 1(N) $\text{K}_2\text{Cr}_2\text{O}_7$ soln.

$\therefore V_1 \times 0.008$ g oxygen

C.O.D. of 50 mL water = $V_1 \times 0.008$ g.

\therefore C.O.D. of 1 litre water = $\frac{V_1 \times 0.008 \times 1000}{50}$ g.